N89-21739

1988

NASA/ASEE SUMMER FACULTY FELLOWSHIP PROGRAM

MARSHALL SPACE FLIGHT CENTER
THE UNIVERSITY OF ALABAMA

CONSTRUCTION OF AN ALTERNATING GRADIENT MAGNETOMETER

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Date:	August 19, 1988
Contract No.:	NGT01-002-099 University of Alabama

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ABSTRACT

A magnetometer is described which was constructed to facilitate the study and characterization of the magnetic properties of high transition temperature superconductors. This instrument has been used to measure the D.C. magnetic susceptibility of several superconducting compounds as a function of temperature.

The construction of the magnetometer and the operating parameters are discussed in detail.

ACKNOWLEDGEMENTS

I would like to thank Dr. Eugene Urban, Dr. Palmer Peters, Mr. Charles Sisk and Mr. James Jolley for the assistance and encouragement which they have provided during the course of this project.

I also wish to thank the ASEE and NASA for providing the opportunity for me to work at MSFC.

LIST OF FIGURES

INTRODUCTION

The magnetic properties of materials form the basis of many practical applications. For this reason the study of these properties is an important part of modern solid state physics. The response of a solid to an external magnetic field is described by the equation

$$B = H + 4\pi M \quad \text{(cgs units)} \qquad (1)$$

H is the strength of the applied field, in Oersteds. B is the magnetic induction within the material , in Gauss, and M is the magnetization. M is a property of the material and in a homogeneous solid it is equal to the average magnetic moment per unit volume. In paramagnetic and diamagnetic homogeneous solids M is directly proportional to H so

$$M = X_m H \qquad (2)$$

where X_m is the magnetic susceptibility. In a paramagnetic substance X_m is positive and in a diamagnetic substance it is negative. For simple homogeneous materials a graph of M vs H yields a straight line of slope X_m.

The alternating gradient magnetometer operates on the principal that a material having magnetization M will experience a force when placed in a spatially varying magnetic field.

$$F = M\,(dH/dx) \qquad (3)$$

M is the component of magnetization in the x-direction and dH/dx is the x-component of the magnetic field gradient. The construction of a gradient magnetometer using an alternating field

$$H = H_o \text{ Sin } wt \qquad (4)$$

has been described by P. J. Flanders[1] The instrument described here is based on that paper.

OBJECTIVES

The objectives of this project were to:

1. Construct an alternating gradient magnetometer capable of performing magnetic susceptibility measurements an small solid samples.

2. Measure the D.C. magnetic susceptibility of high transition temperature superconductors as a function of temperature from room to liquid nitrogen.

THEORY

I. The Magnetometer

An alternating magnetic field of the form $H = H_o \sin wt$, where H_o has a constant gradient dH_o/dx, is produced by a pair of sweep coils. The sample, whose magnetization is to be measured, is placed on the end of a vane which forms a cantilever. The alternating field produces a sinusoidal force on the sample which causes it, and the cantilever, to vibrate at the frequency w. The upper part of the vane is a piezoelectric bimorph which produces a voltage proportional to the deflection of the sample. Since the deflection of the sample is proportional to the applied force and the force is proportional to the magnetization, the piezoelectric voltage is proportional to the magnetization of the sample. If a large direct current field H_{dc} is applied, and if H_{dc} is much larger than H_o, then the D.C. magnetization can be measured as a function of magnetic field.

The sensitivity of the magnetometer is optimized by vibrating the vane at its normal mode frequency, or one of its overtones. Since the vane is vibrating at normal resonance it is very sensitive to external disturbances and must be isolated, as nearly as possible, from outside oscillations.

II. Superconductivity

If an ideal superconductor is placed in an external magnetic field in the x-direction the field will penetrate a small distance into the surface. The internal magnetic induction is given by

$$B = B_o \exp(-x/L) \qquad (5)$$

L, the penetration depth is of the order of 1000 A in pure elemental superconductors. For bulk samples the internal magnetic induction B = 0, or approximately so. The superconductor in this state is a perfect diamagnet with susceptibility

$$X_m = -1/4\pi \tag{6}$$

So the magnetization $M = -H/4\pi$ in an ideal bulk superconductor. A plot of M vs H will yield a straight line having slope X_m. In granular superconductors such as the ones under study at MSFC the magnetic field will penetrate the sample and the flux lines can be "pinned" by impurities and grain boundaries. This leads to hysteresis in the M vs H curves. This effect has been observed by D. Wong et al in the case of small fields[2].

The recent discovery of superconductivity at temperatures above the normal boiling point of liquid nitrogen[3] (77K) has resulted in a frenzy of research activity into the properties of the new materials. Researchers at the Space Science Laboratory have made significant contributions in the field [3 4 5], in collaboration with scientists at The University of Alabama in Huntsville and at Lockheed Missiles & Space Co. Current research has been centered around materials having a nominal composition $R_1Ba_2Cu_3O_x$ + AgO where R is a rare earth ion and x is approximately 6.8 . These materials have shown several unusual magnetic properties which warrent further investigation.

APPARATUS

1. The System

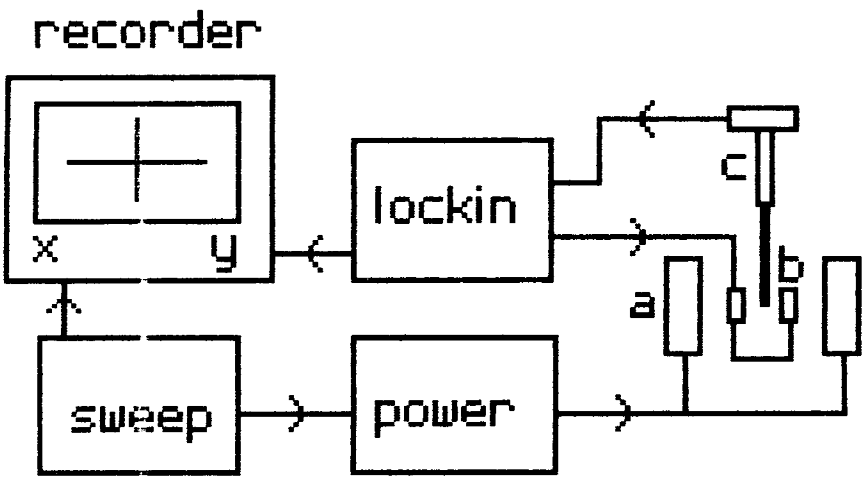
The complete system is composed of (a) Sweep Coils, (b) Field Coils to produce the D.C. field, (c) The Vane and Piezoelectric Transducer, and (d) Electronics. The system is shown in **figure 1.**

The output of a lockin amplifier is used to drive the sweep coils. The sweep coil current was maintained at 150 mA by a 5X power amplifier. The voltage output from the transducer is measured with the lockin amplifier which drives the y-axis of an x-y recorder. A bipolar operational amplifier is used to drive the field coils. The coil current is monitored and plotted on the x-axis of the x-y recorder. The resulting output is a graph of magnetization vs applied field. From this graph the D.C susceptibility can be determined. A more detailed view of the transducer and coils is shown in **figure 2.**

2. The Vane and Transducer

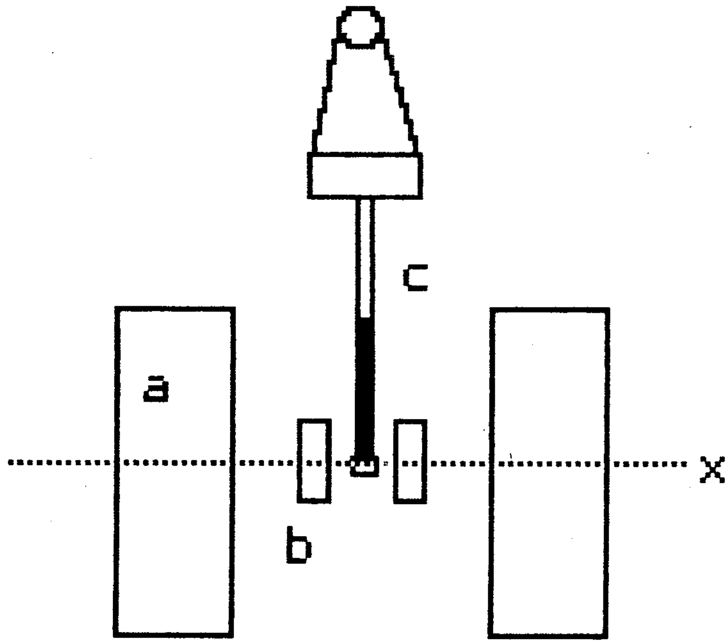
The transducer was constructed using Kynar Piezofilm[6], a polarized polyvinylidine flouride film which was metallized on both sides with aluminum. The complete vane with transducer is shown in **figure 3a.** Strips of Kynar were glued to a phosphor bronze piece with silver varnish to form a piezoelectric bimorph. An extension made from 29 mil thick graphite composite was glued to the end of the bimorph with GE 7031 varnish. The vane extension allowed the sample to be cooled while the Kynar remained at or near room temperature.

The bimorph was clamped between two contacts made from copper foil and the entire assembly was suspended by two rubber bands to provide isolation from external vibrations. A #40 Copper-Constantan (Type K) thermocouple was cemented to the end of the extension with 7031 varnish so that the sample



(a) Field coils. (b) Sweep coils.
(c) vane and Transducer.

Figure 1. Magnetometer System.



(a) Field coils. (b) Sweep coils.
(c) Vane and support.

Figure 2. Detail of Vane and Coils.

temperature could be measured. The sample was attached to the end of the vane extension, in contact with the thermocouple, using silicon grease. Several vanes were constructed having resonant frequencies of 40 to 50 Hz depending on sample mass and, somewhat, on the temperature.

3. Sweep Coils

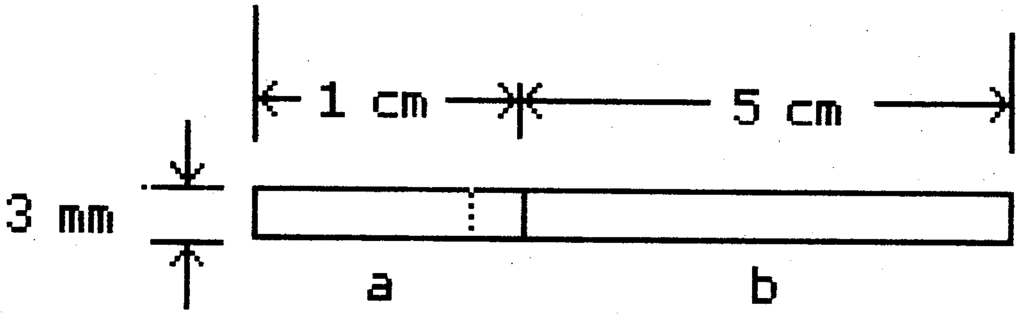
The sweep coils are shown in **figure 3b**. These were rigidly mounted with a separation distance of 6 mm. **Figure 4** shows the magnetic field as a function of distance between the two sweep coils. The current was 100 mA D.C. and the field gradient was 22.1 Oe/cm and can be seen to be quite constant for several mm about the center position. The actual vibrational amplitude of the sample was presumed to be much less than 1 mm since the motion was not visible at any time. The sweep coils were driven at 150 mA during most of the experiments.

4. Field Coils

The field coils were air core solenoids having an outer diameter of 4.75 in, an inner diameter of 2.25 in, and a thickness of 1.50 in. Each coil had a D.C. resistance of 15 Ohms. When placed 6.5 in apart they provided a field of 26 Oe/A. **Figure 5** shows the field profile for these coils. The bipolar operational amplifier used had a maximum voltage output of $\mp$ 15 V giving a maximum field of $\mp$ 52 Oe with the coils in parallel.

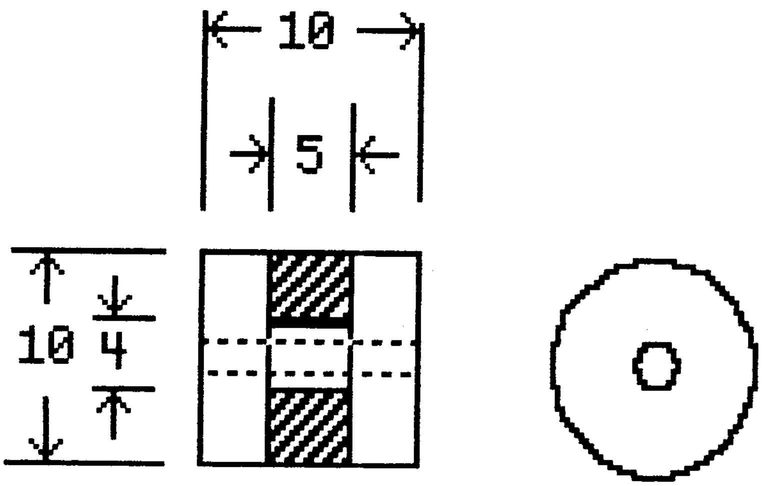
5. Electronics

A lockin amplifier was used for synchronous detection of the transducer signal. The voltages from the two sides of the bimorph were subtracted by the amplifier. This doubled the output voltage while subtracting broadband noise caused by mechanical vibrations and electrical pickup. Much of the pickup noise could be eliminated by driving the vane slightly off resonance and measuring the quadrature (90^{o}) signal. The output from the



(a) Piezoelectric Bimorph
(b) Vane Extension

Figure 3a. Detail of Vane.



Dimensions in mm. Wound with 250 Turns of #36 Formvar insulated copper wire.

Figure 3b. Detail of Sweep Coils.

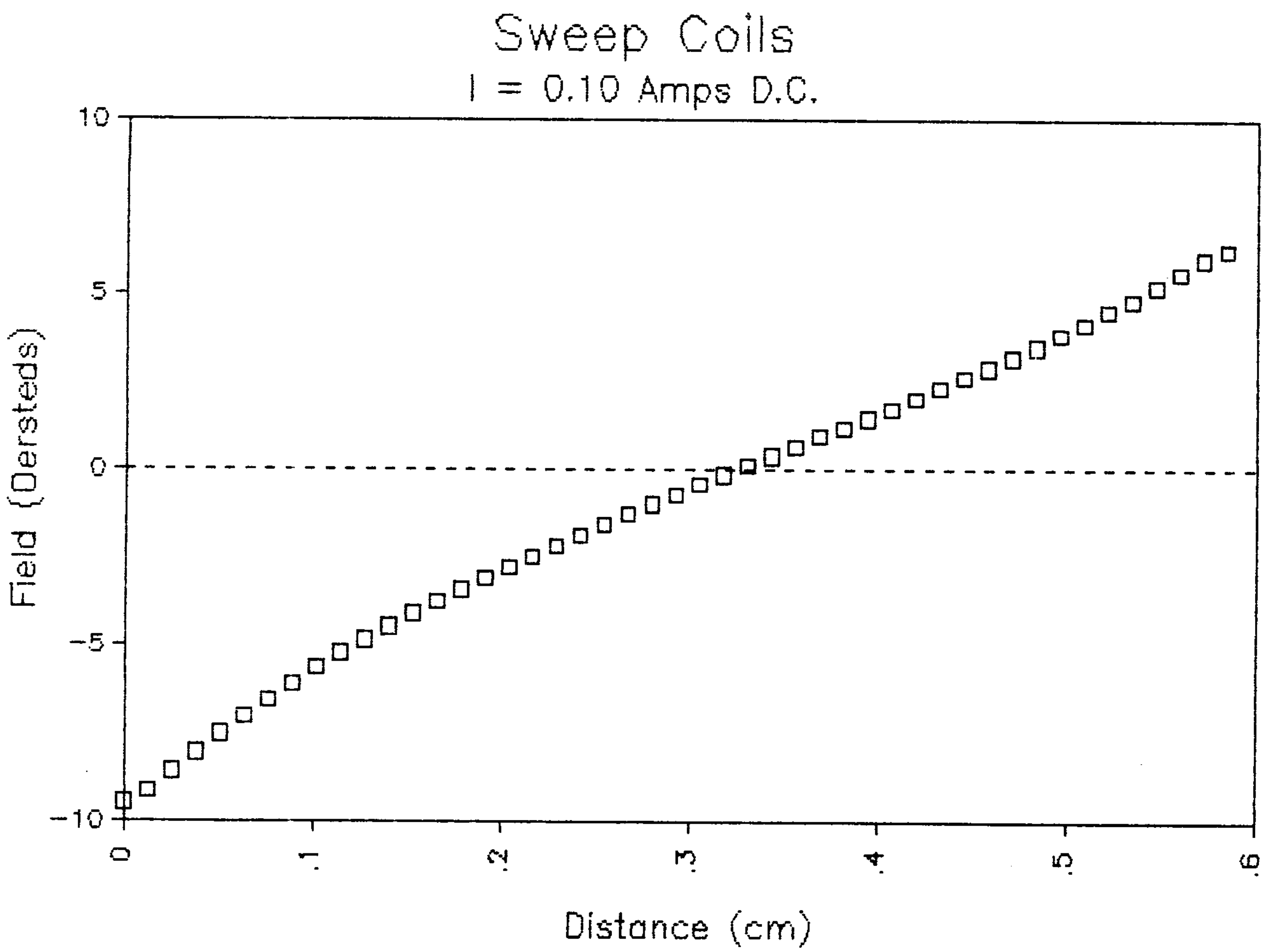


Figure 4. Field Map of Sweep Coils.

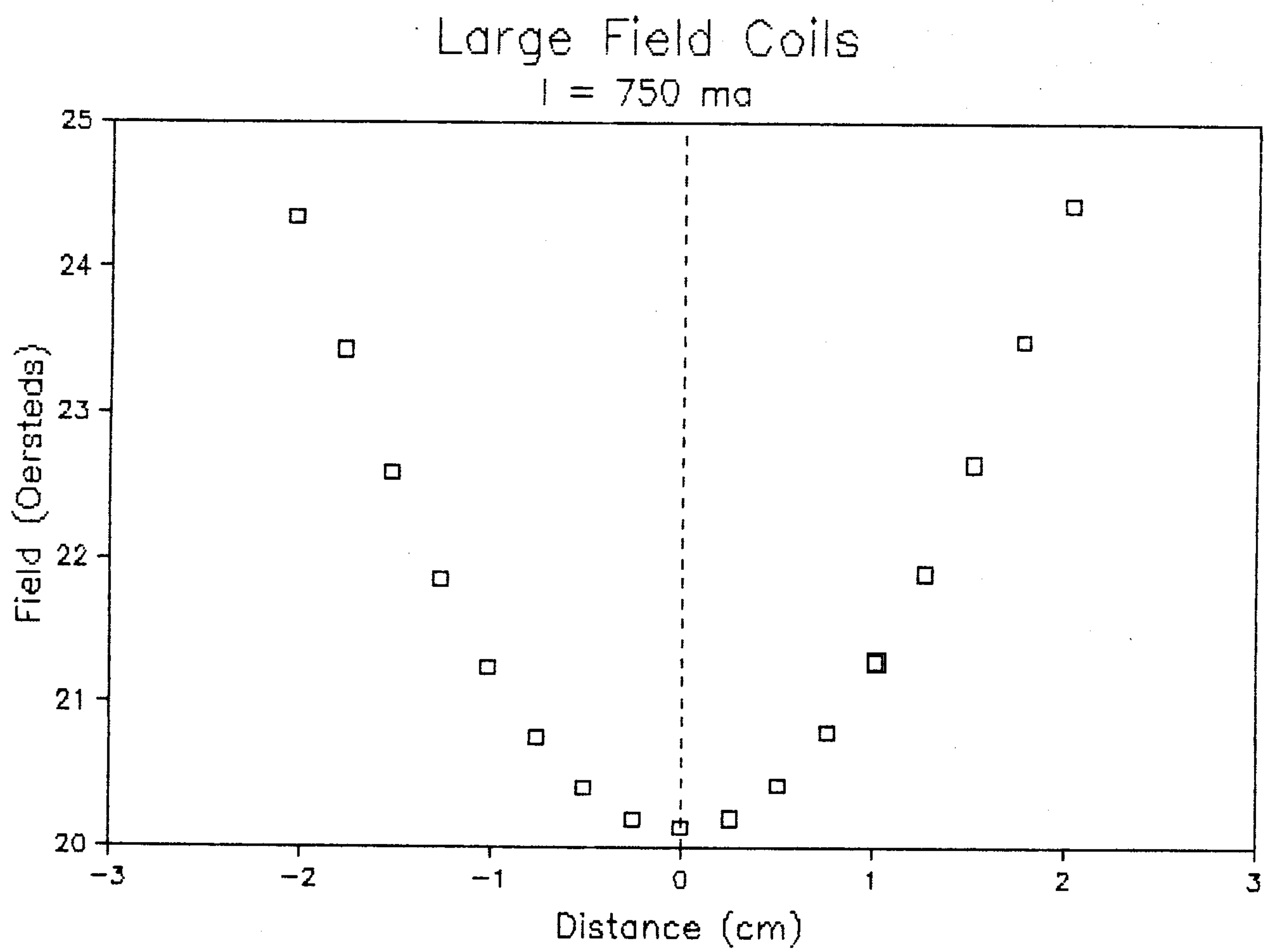


Figure 5. Field Map of Field Coils.

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lockin signal channel was proportional to the sample magnetization. This was recorded on the y-axis of an x-y recorder. The field coil current was passed through a shunt resistor and the voltage from this was recorded on the x-axis of the recorder. The field coil current was swept at a rate of about 100 Oe/min.

In order to measure the magnetization as a function of temperature the sweep coil assembly was submerged in a plastic foam dewar of liquid nitrogen. The sample, on the end of the vane extension, was just above the surface of the liquid. As the liquid evaporated the sample would slowly warm up.

Operation and Experimental Results

1. Magnetometer Calibration

The magnetometer was calibrated using two samples of known susceptibility[7]. (a) A piece of Ni ribbon having a specific susceptibility of 52.38 emu/gm, and (b) A pellet of Nd_2O_3 having a specific susceptibility of 0.0102 emu/gm . Although it is ferromagnetic (X_m is a function of H) the Ni sample gave a linear M vs H curve for the low fields used. From the slope of the M vs H curves the output of the x-y recorder could be calibrated. It was found that each time a particular vane was used it needed to be recalibrated. Presumably this was due to slight changes in vane position associated with handling, which was required to change samples.

2. Thermocouple Calibration

The copper-constantan thermocouple which was used to monitor the sample temperature was calibrated at liquid nitrogen temperature and at the ice point. It was found to be off by -0.004 mV at the liquid nitrogen boiling point and +0.006 mV at the ice point. These errors correspond to 0.24 degrees C and 0.16 degrees C respectively. A thermocouple correction table was generated by taking a linear fit each 10 degrees. This should insure that the temperature measurements were accurate to within 0.1 degrees. The thermocouple correction table is given in the appendix.

3. Experimental Results

Data for a sample of $Sm_1Ba_2Cu_3O_x$ doped with AgO in a ratio of 3:1 is presented in **figures 6 through 10**. Since the material is paramagnetic when in the normal state and diamagnetic in the superconducting state, the superconducting transition is

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Figure 6. Data for SM123+AgO 3:1

Temp (K)	Slope (uV/A)	X (emu/gm)	1/X (gm/emu)
89.5	-5400.00	-6.1020	-.1639
90.5	-5400.00	-6.1020	-.1639
92.8	-5100.00	-5.7630	-.1735
93.1	-4800.00	-5.4240	-.1844
93.7	-4650.00	-5.2545	-.1903
94.5	-4300.00	-4.8590	-.2058
95.2	-4150.00	-4.6895	-.2132
96.7	-3800.00	-4.2940	-.2329
97.75	-3650.00	-4.1245	-.2425
98.5	-3100.00	-3.5030	-.2855
99.5	-2800.00	-3.1640	-.3161
100.1	-2250.00	-2.5425	-.3933
101.2	-1750.00	-1.9775	-.5057
101.7	-1450.00	-1.6385	-.6103
102.5	-1000.00	-1.1300	-.8850
103.6	-275.00	-.3108	-3.2180
105.4	3.25	.0037	272.2941
108.1	3.00	.0034	294.9853
110.6	2.25	.0025	393.3137
111.8	2.00	.0023	442.4779
112.7	2.00	.0023	442.4779
113.9	1.88	.0021	470.7211
116.2	1.83	.0021	483.5824

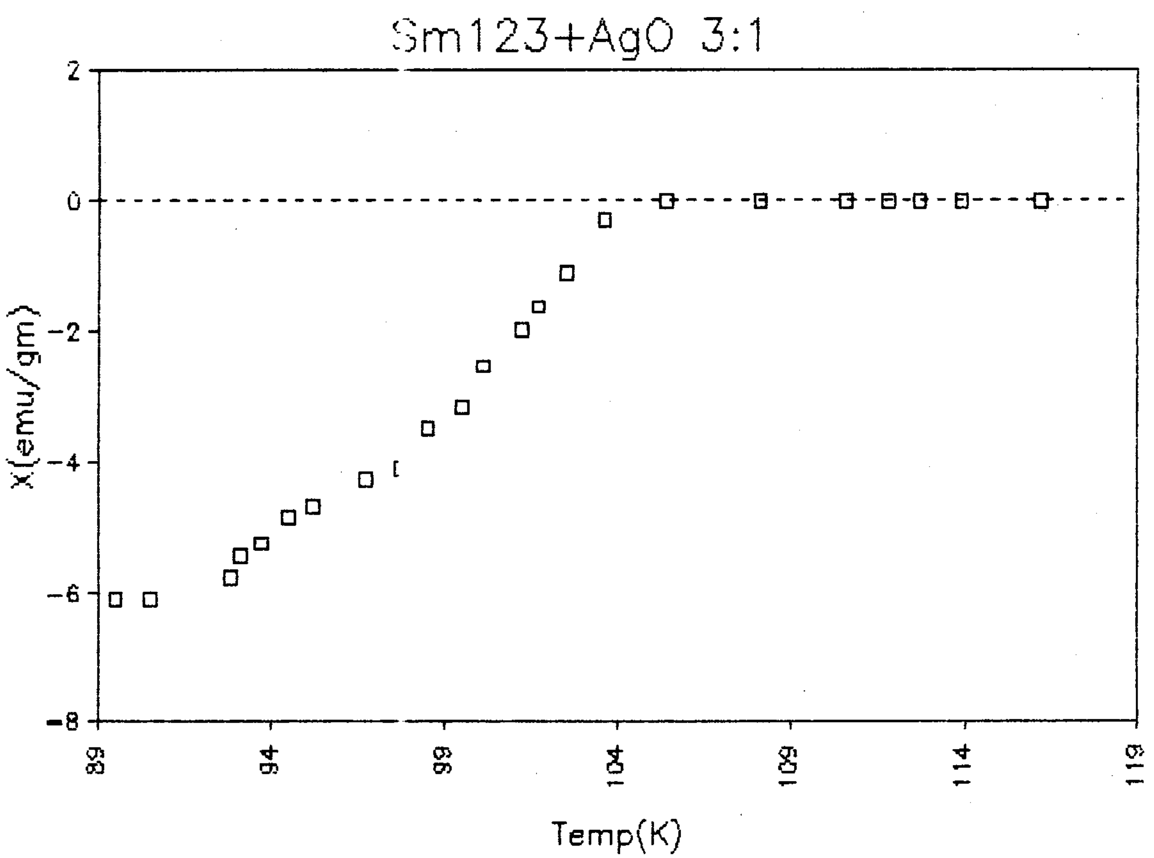

Figure 7. X_m vs T for SM123 + AgO 3:1.

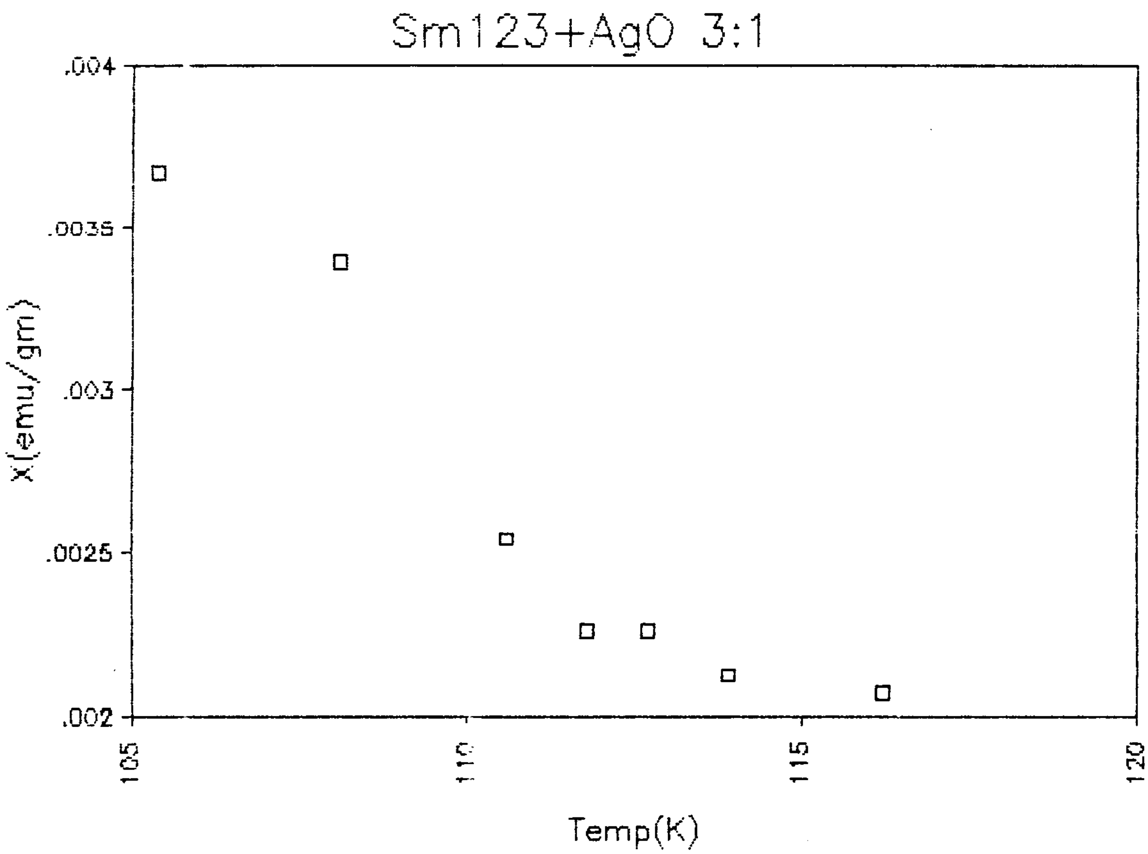

Figure 8. X_m vs T for T > T_c.

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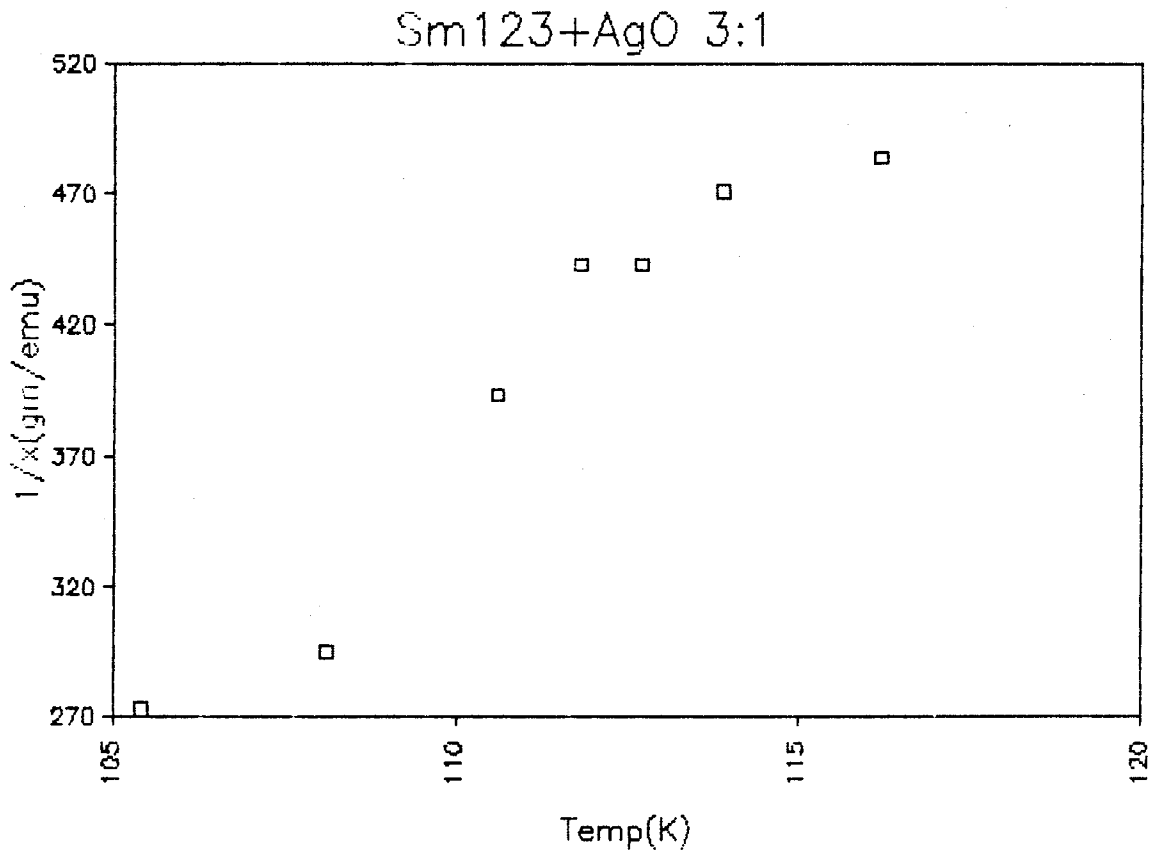

Figure 9. $1/X_m$ vs T for $T > T_c$

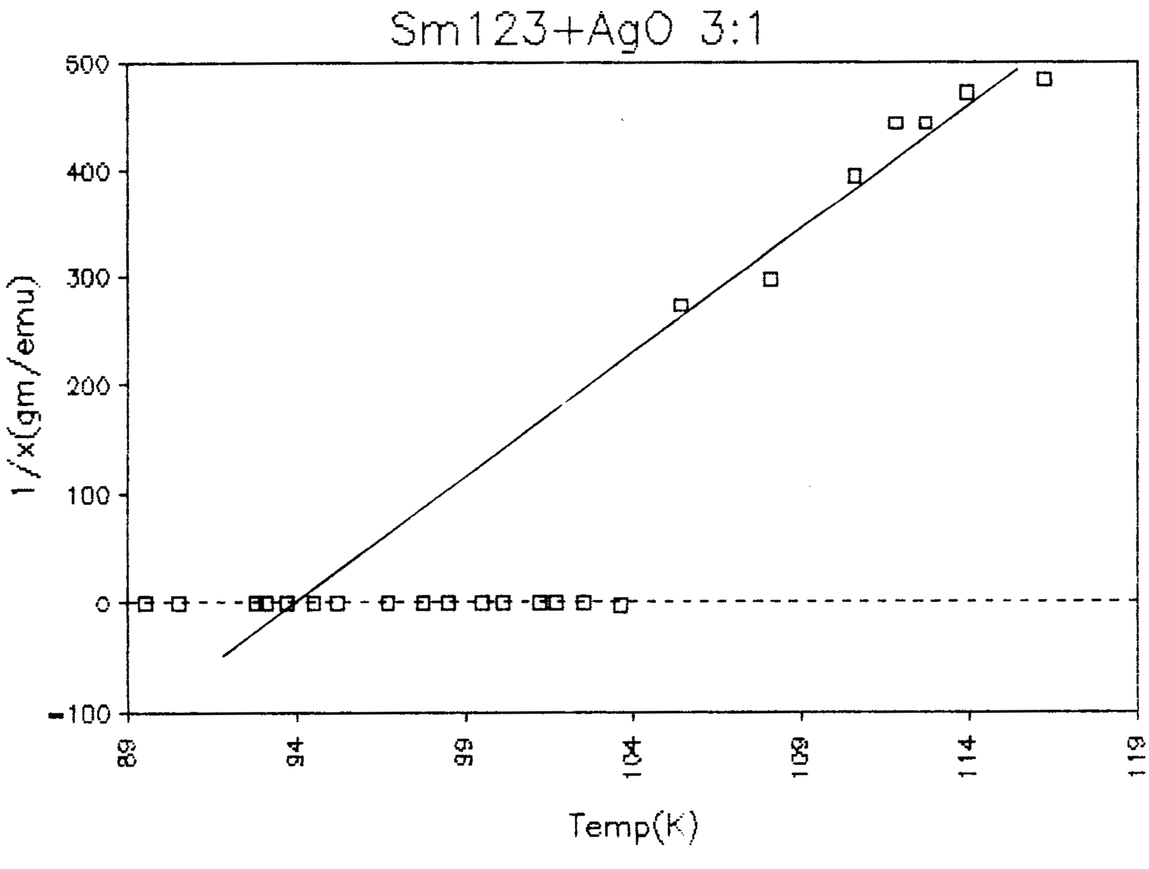

Figure 1Ø. $1/X_m$ vs T

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clearly shown in **figure 7**. The susceptibility begins to go diamagnetic at about 104 to 105 K. This indicates the onset of a superconducting phase. A second phase begins to show up at about 97 K and the sample becomes fully superconducting somewhere between 90 and 92 K. Resistance data on this sample, however, indicate an onset temperature of 97 K and a zero-resistance temperature of 90 K. It is evident that the resistance measurement has missed the initial phase because no complete conducting path, of this phase, existed across the sample. The second, lower temperature phase, did have such a path and it clearly shows up in the resistance data. The seven points having positive magnetization are shown in **figure 8** plotted on a expanded scale. The susceptibility of a paramagnetic material obeys the Curie-Weiss law

$$X_m = C / (T - \Theta) \tag{7}$$

where C is a constant, T is the absolute temperature and Θ is the Curie temperature. A plot of $1/X^m$ vs T should yield a straight line of slope 1/C and intercept Θ/C at $1/X_m = 0$. Figure **9** shows the plot of $1/X_m$ vs T for the points of positive magnetization. The least squares linear fit to these points yields the equation

$$1/X_m = -2102 + 22.5\ T \tag{8}$$

which gives $\Theta = 93.4$ K. Several recent papers on the magnetic properties of these compounds, using Er, Dy, and Ho as the rare earth ions, without the AgO dopant have reported values of Θ which vary from -4 to -7 K [9,10] indicating that they are antiferromagnetic. Although the samples under study here appear to obey the Curie-Weiss law, they are not antiferromagnetic. **Figure 10** shows $1/X_m$ vs T for the complete data set. The intercept at 93.4 K is shown.

CONCLUSIONS AND RECOMMENDATIONS

The alternating gradient magnetometer has proved to be a powerful and versatile tool for the study of the magnetic properties of small samples. The data shown in this report represents one of the ten different compounds which were studied. These compounds were all doped with AgO and all showed an elevated Curie temperature. Furthermore, it has been demonstrated that superconducting phases can be detected which are not seen in electric resistance studies.

Three recommendations which will increase the range and usefullness of this instrument are:

1. A dewar needs to be built which will provide better temperature control during the course of the measurements.

2. A larger D.C. magnet is needed to extend the range of measurements up to several kilogauss.

3. Isolation from outside vibrations needs to be improved. This should extend the lower limit of measurement to 10^{-4} emu/gm or less.

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APPENDIX

Thermocouple Correction Table

T(C)	Emf(Tab)	Emf(Corr)	dE	<dE/dT>	<dT/dE>
0	0	.006			
-10	-.383	-.377	.3826	.0378	26.445
-20	-.757	-.750	.3736	.0369	27.127
-30	-1.121	-1.114	.3636	.0359	27.883
-40	-1.475	-1.468	.3536	.0349	28.683
-50	-1.819	-1.811	.3436	.0338	29.573
-60	-2.152	-2.144	.3326	.0328	30.521
-70	-2.475	-2.467	.3226	.0318	31.482
-80	-2.788	-2.779	.3126	.0307	32.611
-90	-3.089	-3.080	.3006	.0295	33.939
-100	-3.378	-3.368	.2886	.0283	35.318
-110	-3.656	-3.646	.2776	.0272	36.745
-120	-3.923	-3.913	.2666	.0260	38.440
-130	-4.177	-4.166	.2536	.0248	40.381
-140	-4.419	-4.408	.2416	.0235	42.527
-150	-4.648	-4.637	.2286	.0223	44.915
-160	-4.865	-4.853	.2166	.0210	47.587
-170	-5.069	-5.057	.2036	.0198	50.596
-180	-5.261	-5.249	.1916	.0185	54.159
-190	-5.439	-5.426	.1776	.0171	58.602
-200	-5.603	-5.590	.1636	.0164	61.109

$T = To + \langle dT/dE \rangle (E - Eo)$